Asian Journal of Chemistry Vol. 13, No. 4 (2001), 1305–1311

Study of Chelating Ion Exchange Resin Containing 8-Hydroxy Quinoline and Separation of Metal Ions by Selective Sorption on the Resin Column

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Chelating ion exchange resin was synthesised from oxine (8-hydroxy quinoline)-resorcinol-formaldehyde. The physico-chemical properties like % moisture content, true density of resin (d_{res}), apparent density of resin (d_{col}), void volume fraction, total exchange capacity, rate of exchange, interruption test, salt splitting capacity, thermal stability, resin stability, pH study for metal ions, effect of metal ion concentration on exchange capacity were measured. The resin was characterised by IR and TGA. The quantitative separation of copper-nickel, zinc-nickel was accomplished by selective sorption on column.

INTRODUCTION

The most important ion exchange resins produced and employed today are synthetic resins. The ion exchange resin structure possesses a maximum resistance to oxidation, reduction, mechanical wear and breakage and are insoluble in common solvents. The ion active group which is fixed to high molecular weight polymer backbone is immobile. The electrical charge of the ion active group is always balanced by an equivalent number of oppositely charged ions, which are mobile and can exchange with other ions of a similar charge. The nature of the ionizable groups attached to the hydrocarbon network determines the chemical behaviour of an ion exchange resin.

Chelating resins are such synthetic ion exchange material which possess the combined advantageous properties of chelating groups and polymers. Therefore in recent years chelating resins have evoked considerable interest of analytical chemists and metallurgists. There has been considerable activity in the field of chelating ion exchangers in recent years. Studied on ion exchangers and organic or inorganic ligands have led scientists to develop ion exchange polymers possessing chelating properties. In analytical as well as in preparative inorganic chemistry there exists a need for chelating polymers, which combine the ease of operation of conventional ion exchangers with specificity or selectivity of the ligand.

A wide range of chelating ion exchange resins has been prepared from different monomers containing 8-hydroxy quinoline and related monomers[1, 2]. Ion exchange resins have proved to be of great importance due to its applications in water conditioning, water softening and deionization. There are many useful reports on ion exchange separations methods in chemical processes[3].

EXPERIMENTAL

Preparation of the Resin: 8-Hydroxy quinoline AR grade ground to a fine powder (0.1 mole) was added to 47.5 mL of sodium methoxide and stirred to give yellow paste. Formaldehyde (0.2 mole) as 37% formalin was added and stirred until red solution was obtained.

A solution of resorcinol 0.1 mole in sodium methoxide was added to the oxine-formaldehyde solution. The mixture was heated on a water bath with constant stirring until a viscous solution was obtained. The resin mixture was poured into 250 mL of liquid paraffin at 80°C and stirred sufficiently to produce small droplets gelled to resin. The resin was filtered from the liquid paraffin and rinsed with chloroform, washed thoroughly with soap solution to remove all paraffin. The resin was washed with methanol and finally with water to remove monomer impurities[4]. The resin was stored in fully swollen form for further studies.

RESULTS AND DISCUSSION

The different physico-chemical properties observed were as follows:

TABLE-1
PHYSICO-CHEMICAL PROPERTIES OF RESIN

Moisture content %	19.96
Water regain capacity	0.199 (gg^{-1})
Solid %	80.04
True density	1.24 g/cc
Apparent density	0.5 g/cc
Void volume fraction	0.59
Concentration of fixed ionogenic group	5.95
Sodium exchange capacity	6.0 mmole/g dry resin

The synthesized resin was characterized by IR and TGA methods.

The absence of 1,3-disubstituted band of resorcinol at 1650 cm^{-1} and 1500 cm^{-1} confirms the polymerization of the monomer.

The thermogram of resin (Fig. 1) shows a single step degradation[5]. The decompositions involve 41.36% weight loss within the temperature of 685°C. From this it confirms that the resin sample possesses high thermal stability.

The effect of pH on sodium exchange capacity showed that the resin exhibits good cation exchange capacity above pH 12.0. Thus the cation exchange capacity is pH dependent (Fig. 2).

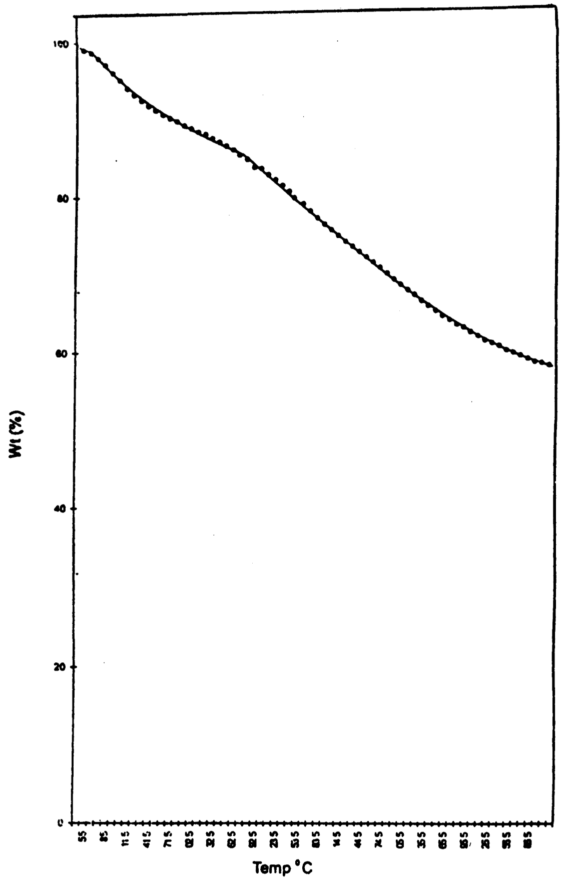

Fig. 1. Thermogram of resin.

Fig. 3 shows faster rate of exchange in the beginning which is attributed to the law of mass action. Gradually the rate of diffusion of ions towards the interior of the resin shows slow exchange due to shell diffusion or particle diffusion which was observed in rate of exchange process.

The effects of pH on metal ions (Fig. 4) show the maximum chelating capacity for copper(II) and lead(II) which was found to be at pH 5.0, for zinc(II) it was found at pH 6.0 and for nickel(II) it was not maximized. The exchange capacity remained constant from pH 1–7 in the case of nickel(II).

The exchange of metal ions on resin exhibited the time dependent phenomenon. 50% of metal exchange with resin for copper(II), zinc(II) and nickel(II) was within 1 h and for lead(II) it was after 4.5 h (Fig. 5).

Interruption test for metal ions exhibit that the rate of exchange of metal ions has direct relation to its particle diffusion process (Fig. 6).

Fig. 7 shows that the uptake of metal ions by the resin increases in proportion

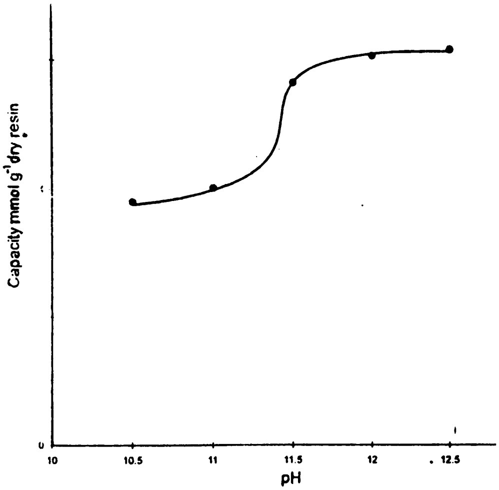

Fig. 2. Effect of pH on sodium exchange capacity.

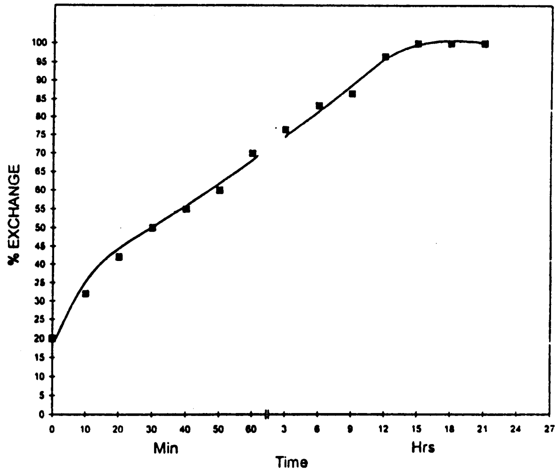

Fig. 3. Rate of exchange for sodium ion.

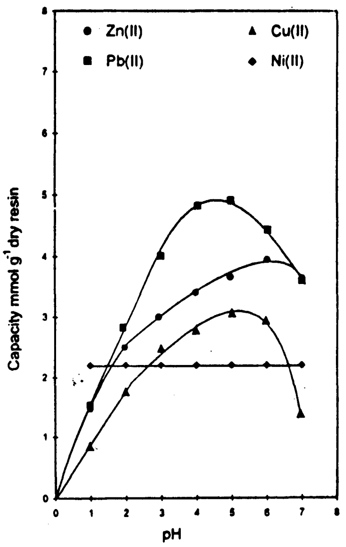

Fig. 4. Effect of pH on metal ion capacity.

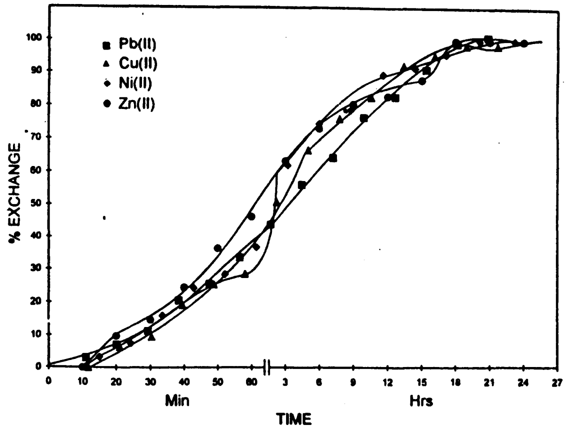

Fig. 5. Rate exchange for metal ion.

to the concentration of metal ion solution. Saturation of the resin takes place at 0.2 M concentration for copper(II) and nickel(II), at 0.25 M for lead(II) and at 0.3 M for zinc(II). It is observed that the amount of solution in the sorbent increases with increasing concentration of external solution.

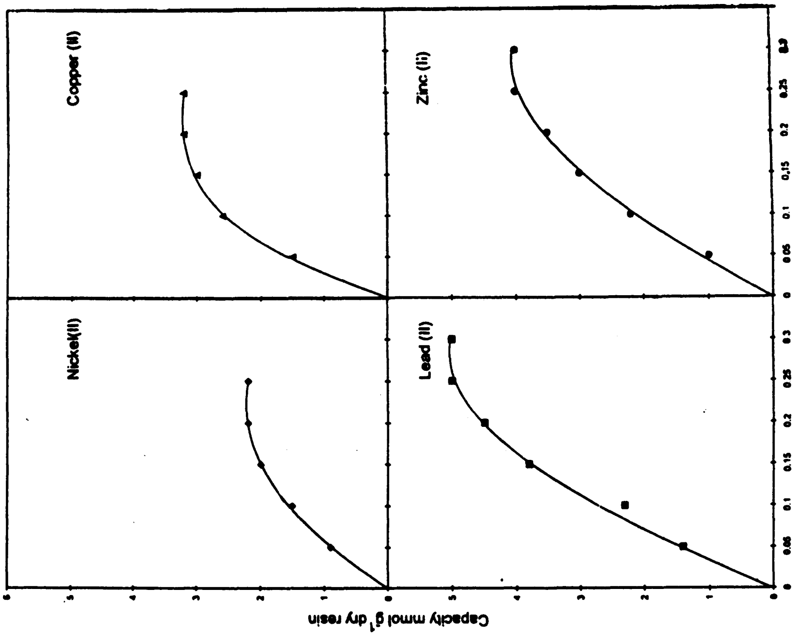

Fig. 7. Effect of metal ion concentration.

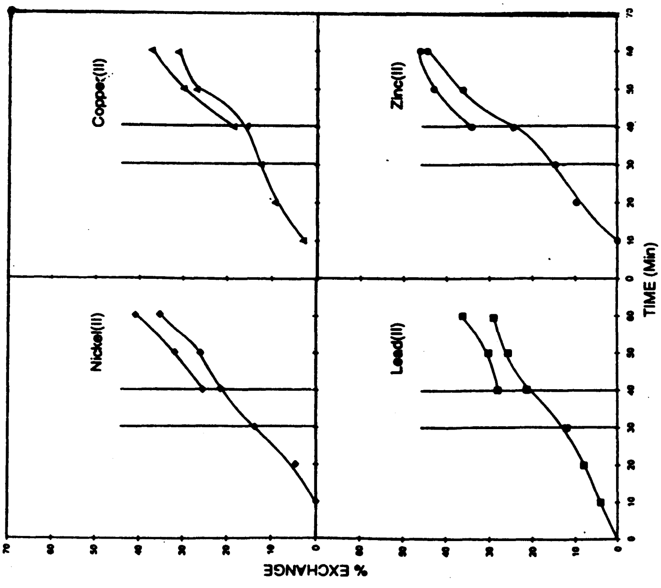

Fig. 6 Interruption test for metal ions.

Separation of Copper(II) from Nickel(II) and of Zinc(II) from Nickel(II) by Selective Sorption on Column

Selective sorption[3, 6] on resin column were carried out for equimolar mixture of copper(II) and nickel(II) and also zinc(II) and nickel(II), copper(II) and zinc(II) being strongly absorbed on the column at pH 5.0 and pH 6.0 respectively, while nickel(II) moving down to column. 2 M HNO_3 was used as eluting agent. The recovery of copper(II) was 80% and of zinc(II) was 92%.

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(*Received:* 12 February 2001; *Accepted:* 28 May 2001) AJC-2372